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Overview

In this quarter we have succeeded in transferring high growth rate diamond process from our 8kW prototype to our 75kW reactor, and generated the first free-standing diamond films of greater than 4"x4" size. Our efforts have concentrated on: improving the growth rate and film quality of the diamond growth process, exploring the operating envelope of the 75kW reactor, and designing improvements to fully realize the potential of the large scale high growth system. New program components have been initiated for developing release techniques on large substrates, improving film flatness, and improving uniformity of thickness and morphology. A thermal conductivity test stand is in the planning stages for in-house testing of samples in the near future.

75kW Reactor

Reactor operations have become routine, with regular 24 hour operation, although at reduced power (40-45kW). Over two thousand hours of transmitter time have been logged for MCM process development.

We have completed the design and fabrication of a modified substrate mounting and cooling stage, which will improve the heat removal capability and uniformity over the existing design. The new design makes use of a novel method of maintaining good cooling uniformity under the mechanical and thermal stresses present in the reactor, and should allow operation beyond the rated 75kW of the transmitter. Pre-assembly of the stage has been completed, and the installation should be complete and operations resumed by mid February 1994.

During the reactor shutdown period for the stage installation, we are also scheduling upgrades to the 75kW transmitter and transmission line. These involve both software changes to the programming logic of the transmitter controller (improved safety interlocks and modified logic for extending the magnetron filament lifetime) as well as hardware changes to the transmission line to reduce reflected power and optimize the use of transmitter power.

Process Transfer, Ongoing Experiments

We have continued to develop diamond growth process in conventional (H_2, CH_4, O_2) chemistries from our prototype to the 75kW reactor. We have found that operation at 40kW (30-35kW forward power) yields throughput to the 300-400 mg/hr range, consistent with the prototype projections and the 75kW program plan. Following this curve, we should be able to obtain the 1 g/hr goal with 75kW operation.

We have found that we can strongly affect the uniformity of the deposition morphology and thickness without sacrificing growth rate. We can typically obtain 10-15% thickness uniformity over 6" to 8" diameter depositions, however morphology uniformity has been more difficult to obtain. The morphology appears to depend critically on the substrate temperature, and thus is expected to improve markedly with the stage upgrade schedule for Jan/Feb 94.

We have undertaken to develop a technique for releasing the diamond film from the substrate after deposition without necessitating a wet-etch of the substrate. In addition to eliminating the use of hazardous chemicals, this further reduces the cost of the diamond film by eliminating the use of expendable substrates. Furthermore, our experience suggests that a significant portion of the wafer bow is due to the thermal expansion mismatch between the substrate and the film, and that this bowing is relieved during cool down with a substrate which releases the film. Successful substrate releases have been run in the prototype reactor on molybdenum substrates up to 3" diameter, and experiments are underway to test other materials and larger diameters as well.

We have completed a series of experiments designed to test the effect of CH_x to C_2H_x radical conversion in high temperature and long residence time (diffusive) cases. We used a nozzle arrangement to inject CH_4 directly to the substrate surface at near-sonic speeds. If acetylene conversion in long residence time is a factor, this should have shown marked improvement in growth rate, and local asymmetries in morphology corresponding to the injection geometry. We have been unable to observe any of these effects, so our preliminary conclusion is that either the CH_4 injection must be more localized to overcome convective effects, or more likely, that C_2H_x conversion is not a serious limitation to diamond growth.

We have undertaken some runs in unconventional chemistry, notably very low flow mixtures of CH_4 and CO_2. As in our prototype experiments, we have been able to achieve good growth rates with these mixtures, however the material quality was poor. Our experience with these mixtures shows that they perform best at lower substrate temperatures, which have been difficult to obtain with the present stage. These experiments will resume after the stage rebuild is complete.

We have arranged to purchase the design of an existing thermal conductivity test stand. The use of an existing and proven design will allow faster time to full operation and utility in the MCM program, and better bench marking with other measurement techniques.

Reactor Prototype Related Studies

We are continuing to study high growth rate (HGR) process in our PDS19 reactor, the prototype for the 75kW system, operating at 8kW and 2.45 GHz. Our recent efforts have been in studying the path for very high uniformity depositions on substrate sizes which scale up to >6" on the 75kW system. We have identified several key issues in obtaining high uniformity; including alignment of the microwave applicator, position of the substrate on the stage, and (most importantly) uniform temperature of the substrate.

We have also undertaken a series of experiments to determine the limits to high growth operation. A series of runs at high pressure showed that growth rate can increase markedly as power density increases. Further experiments will have to determine the tradeoff of higher power operation with film stress and thermal conductivity. The transfer of these experiments to the 75kW reactor will be a high priority for the next campaign of operations.

Reactor Modeling

The 2D modeling, using the GEM code, has been used quite extensively in the design of the stage rebuild. Although the present version of the 2D code considers only the hydrogen chemistry (with self consistent plasma and microwave effects), the diamond growth and temperature profile are considered to be proportional to the atomic hydrogen flux on the substrate. The profile of this flux generally matches the observed temperature gradient of the substrates when radial heat transfer effects are accounted for.

An extensive series of modeling runs was undertaken to improve the uniformity of the deposition profile by reducing the peaking effect of the atomic hydrogen flux. We did not observe cavity shape improvements dramatic enough to warrant a major rebuild of the reactor vessel, but instead identified conditions which will optimize the uniformity of the deposition using the existing chamber.

The success of the 2D code has been used to model possible alternate reactor geometries for future development. Several such systems are of great interest:

A compact 75kW system, with a single mode cavity, lower cost, and reduced substrate diameter, A modified reactor geometry with higher plasma area utilization,

A larger scale system at lower frequency (around 250 MHz) and several hundred kW microwave power.

The continuing efforts on modeling are concentrating on further understanding and improving process on the 75kW system. As a support tool, the modeling is generally helping to determine ideal substrate mounting configurations and run conditions.